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REPORT

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COUNTRY

USSR

DATE OF
INFORMATION 1951

SUBJECT

Scientific - Chemistry, hydrogenation,
acetylene derivatives

HOW

PUBLISHED

Monthly period

DATE DIST. 25 Jan 1952

WHERE

PUBLISHED

Moscow

NO. OF PAGES 10

DATE

PUBLISHED

Apr 1951

LANGUAGE

Russian

SUPPLEMENT TO
REPORT NO.

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Zhurnal fizicheskoy khimii. Vol XXV, No 4, 1951, pp 495-503,

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KINETICS OF THE HYDROGENATION
OF DIMETHYLACETYLENYL CARBINOL
ON A SKELETON NICKEL CATALYST

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Submitted 3 May 1950

[Figures referred to are appended.]

The system in which liquid-phase catalytic hydrogenation takes place is composed of an unsaturated compound, a solvent, the catalyst, and gaseous hydrogen. So that hydrogenation may take place, both hydrogen and the unsaturated compound must diffuse to the surface of the catalyst, must be adsorbed by it, must react, and then the reaction products must be desorbed.

It is often the case with reactions which take place in the liquid phase that this whole complex catalytic process reflects, as a whole, the relationships governing the diffusion of reagents from a volume to the surface of a catalyst, and the total rate of the process under these conditions is equal to the rate of diffusion. In such a case the reaction will proceed in the "diffusion" region (1).

If diffusion is ignored, it is possible to study the kinetics of the reaction by itself and to determine the so-called kinetic region of the reaction, where the reaction rate reflects directly the kinetics on the surface of the catalyst (1).

Therefore, a study of the actual kinetics and mechanism of any given reaction requires that the diffusion processes be eliminated, which can be achieved by varying the rate of agitation, temperature, quantity of the substance to be hydrogenated, etc.

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Research on hydrogenation processes from the viewpoint of delimiting the diffusion and kinetic regions of a reaction was conducted in the works of S. Yu. Yelovich, G. M. Zhabrova, and V. I. Gol'danskiy (2-4) and in those of D. V. Sokol'skiy and his associates (5-7). In one of their first works (2) Yelovich and Zhabrova studied the kinetics of the hydrogenation of styrene in a solution of glacial acetic acid in the presence of BaSO_4 as catalyst. The rate of hydrogenation of styrene obeyed an equation of the zero order, and the zero order was preserved for a variation of the number of shakings between 100 and 600 per min. It was shown that a low intensity of agitation, from 100 to 490 shakings per min, the hydrogenation reaction took place in the diffusion region in respect to hydrogen, but in the kinetic in respect to the unsaturated compound. At a higher intensity of agitation (over 490 shakings per min) the reaction rate did not depend on the intensity of agitation.

In the kinetic region, the apparent energy of activation, calculated graphically in the temperature interval of 20-100°C, was equal to 5,000 \pm 1,000 cal/mole, and the reaction rate was directly proportional to the quantity of catalyst when the latter was varied from 0.0018 to 0.036 g, on the basis of metallic Pt.

Further study of the influence of hydrodynamic factors on the rate of hydrogenation of unsaturated organic compounds was reflected in the work of V. I. Gol'danskiy and S. Yu. Yelovich (3), devoted to the hydrogenation of oleic acid in a solution of glacial acetic acid in the presence of Pt on BaSO_4 . In this work it was demonstrated that the position of the boundary between the diffusion and kinetic regions depends on the temperature and quantity of catalyst.

In the kinetic region, the reaction rate is proportional to the quantity of catalyst, while in the diffusion region, it approaches a limiting value. [] a similar dependence for the hydrogenation of trimethylethylene in the presence of platinum oxide in a solution composed of a mixture of 95% ethyl alcohol and 5% methyl alcohol, as did D. V. Sokol'skiy and his associates (5-7) for the hydrogenation of cinnamic acid in a solution of 96% ethyl alcohol on a skeleton nickel catalyst and for the decomposition of diazonium salts of metallic copper.

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The present investigation, on the example of the hydrogenation of dimethylacetylenylcarbinol in the presence of a skeleton nickel catalyst, purposes to study the effect of the rate of agitation on the reaction rate and to determine how this effect depends on changes of temperature, quantity of the hydrogenated substance, and quantity of catalyst.

Experimental Part

The process of the hydrogenation of dimethylacetylenylcarbinol in the liquid phase was carried out in the device pictured in Figure 1. The basic components of this apparatus were a gas burette, fitted with a water jacket for maintenance of a constant temperature, and the hydrogenation device proper, or "duck." The duck was placed in a constant-temperature bath of 0.1-cu-m vol and fastened in openings in the rod with the help of a brass brace. The bath was filled with water and kept the temperature constant within the limits of $\pm 0.1^\circ\text{C}$.

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The reaction vessel, the duck, was subjected to strong, uniform shaking with the aid of a 0.25-kw electric motor. The design of the agitation apparatus allowed for a variation of the number of oscillations of the duck between 200 and 1,600 complete cycles per min. The distance of oscillation of the duck was 5.2 cm in all experiments. The number of oscillations was registered with a tachometer. The measuring part of the apparatus was a gas burette with an equalizing flask filled with water. Hydrogenation was carried out with pure electrolytic hydrogen which, for removal of traces of moisture and oxygen, was passed through drying columns filled with calcium chloride and sodium hydroxide, as well as through an incandescent porcelain tube filled with pieces of copper or palladium-coated asbestos.

Dimethylacetylenylcarbinol was characterized by the following constants:

$T_k = 103-104^\circ\text{C}$ at 693 mm,

$\frac{n_D^{21}}{D} = 1.4180$, $d_4^{20} = 0.8613$.

The solvent, was 96% ethyl alcohol, which was first distilled with the use of a dephlegmator. The skeleton nickel catalyst was prepared by leaching the aluminum from a Ni-Al alloy (30% Ni and 70% Al).

The active catalyst was prepared from the alloy by the following method: 1 g of finely ground Ni-Al alloy ($d = 0.5-1$ mm) was placed in a small test tube and inserted in a Kjeldahl flask containing 80 cc of 20% NaOH; the latter was first heated on a boiling water bath. The leaching was conducted on a boiling water bath for 2 hr, after which the alkali was decanted and the resultant catalyst washed four times with distilled water (100 cc at a time) and then twice more with 96% ethyl alcohol (10 cm at a time). A portion of the light particles of the catalyst was lost by decantation in the washing, thus lowering its activity. To preclude loss of the catalyst, all wash water was set aside and allowed to stand, the particles which settled in time being added to the total catalytic mass.

Reagents were added to the reaction vessel (duck) in the following order. To the catalyst, which had been transferred into the duck, were added 20 cc of 96% ethyl alcohol. The temperature required for the experiment was established in the constant-temperature bath 0°C was obtained by filling the bath with ice). Then the air in the reaction system was displaced by a current of hydrogen by means of passing through the system 600-800 cm^3 of H_2 , a test for purity of the hydrogen.

The next operation was preliminary saturation of the catalyst with hydrogen, which was necessary to bring the catalyst to the standard state. The catalyst was agitated for 15 min at a rate of 1,080 oscillations/min. In this period 2-5 cm^3 of hydrogen were absorbed. After saturation of the catalyst with hydrogen, the substance to be hydrogenated and 5 cm^3 more ethyl alcohol were introduced into the current of hydrogen. The moment the motor for producing uniform shaking of the duck was switched on was taken as the beginning of hydrogenation. Then, a reading of the consumption of hydrogen (from the burette) during one minute was taken. The termination of hydrogenation was determined from an absence of change in the water level in the burette over several minutes.

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Results of Experiments

The hydrogenation of dimethylacetylenylcarbinol proceeds according to the following equation:

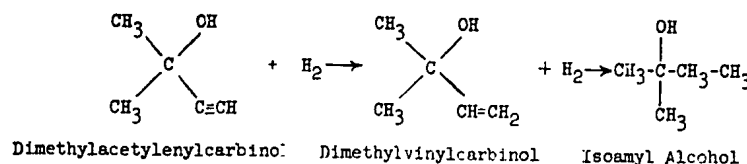


Figure 2 shows the curves which indicate the course of the dimethylacetylenylcarbinol hydrogenation reaction with time when $t_{\text{expt}} = 0^\circ\text{C}$. The axis of abscissae of these curves represents the total volume of hydrogen entering into the reaction, and the axis of ordinates, the rate of the reaction in cm^3/min . From Figure 2 it is evident that at 0°C the reaction is satisfactorily described by an equation of the zero order, since the rate of hydrogenation of dimethylacetylenylcarbinol, expressed in cm^3/min , was constant up to the point at which two thirds of the required hydrogen was absorbed (according to calculation, under normal conditions for the hydrogenation of 0.1723 g of dimethylacetylenylcarbinol into isoamyl alcohol, 91.9 cm^3 are required). No discontinuity corresponding to the conversion of the triple bond to double was observed. Figure 2 also shows the reproducibility of experiments conducted under completely analogous conditions.

When $t_{\text{expt}} = 25^\circ\text{C}$, the reaction is also described by an equation of the zero order. After the order of the reaction had been established, a study made of the effect of the rate of agitation of the reaction mixture on the rate of hydrogenation of dimethylacetylenylcarbinol at 0° and 25°C . The conditions of hydrogenation for the experiments shown in Figure 3 were as follows: 0.1723 g of dimethylacetylenylcarbinol, 0.3 g of the Ni catalyst, and 25 ml of 96% ethyl alcohol as solvent. The results of these experiments can be presented in the form of curves showing the effect of the rate of agitation (oscillations/min) on the rate of hydrogenation of dimethylacetylenylcarbinol (ΔV_0 cm^3/min) (Figure 3). From the curves, it is evident that when dimethylacetylenylcarbinol is hydrogenated at 0° and the intensity of agitation is increased (210-520 oscillations/min), the rate of hydrogenation rises, i.e., the reaction takes place in the diffusion region. Above 520 oscillations/min, the rate constant remains constant, and the reaction takes place in the kinetic region. When the temperature of the experiment is 25°C , the transition from the diffusion to the kinetic region is observed at higher rates of agitation (580 oscillations/min).

When the temperature of the experiment is 25°C , the hydrogenation reaction proceeds more rapidly than at 0°C . An increase in the reaction rate under the given conditions leads in turn to the development of hindrances to sufficiently rapid movement of hydrogen molecules toward the surface of the catalyst, since the rate of the reaction increases much faster when the temperature is increased than the rate of diffusion. The hindrances to hydrogen diffusion are overcome by more intensive agitation of the reaction mixture (then, the reaction proceeds in the kinetic region).

On the basis of the cited data it is possible to calculate the apparent activation energy at different points of the diffusion regions and in the kinetic region.

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When the intensity of agitation is equal to 210 oscillations/min and the temperature of the experiment is 0°C, the rate of hydrogenation of dimethylacetylenylcarbinol, expressed in cm³/min, is equal to 6 cm³/min, while at 25°C and the same rate of agitation, the rate of hydrogenation is equal to 10.1 cm³/min; the apparent activation energy, calculated between these points on the curves (Figure 3) by the formula of Arrhenius:

$$\ln \frac{k_2}{k_1} = \frac{E}{R} \left(\frac{1}{T_1} - \frac{1}{T_2} \right),$$

is equal to 2,961 cal/mole. The apparent energy of activation calculated at a rate of agitation of 330 oscillations/min is equal to 3,477 cal/mole, and at 400 oscillations/min, 3,747 cal/mole. Over 580 oscillations/min, the activation energy remains constant, equal to 4,733 cal/mole. With an increase of the intensity of agitation within the limits studied by us of 210-580 oscillations/min, the apparent energy of activation rises linearly as the rate of agitation increases, but then, with any further increase in the rate of agitation in the range of 580-1,080 oscillations/min, no increase in the apparent activation energy takes place: it remains constant, equal to 4,733 cal/mole.

To study the effect of the rate of agitation on the rate of hydrogenation in dependence on the concentration of the substance to be hydrogenated, a series of experiments was conducted at 0°C. The quantity of dimethylacetylenylcarbinol taken in each experiment of this series was 0.5168 g, i.e., three times as much as in the preceding series of experiments. Again in this case, the reaction obeyed an equation of the zero order up to the point at which two thirds of the theoretically required hydrogen was absorbed.

The results of the second series of experiments, conducted to study the effect of intensity of agitation on the rate of hydrogenation of dimethylacetylenylcarbinol, are shown in Figure 4.

From Figure 4 it is evident that the rate constant for the hydrogenation of 0.5168 g of dimethylacetylenylcarbinol increases continuously as the intensity of agitation rises from 204 to 502 oscillations/min. From 520-1,080 oscillations/min, it remains constant, independent of further increases in the rate of agitation.

The rate constant values for hydrogenation of both 0.1726 g and 0.5168 g of dimethylacetylenylcarbinol for identical rates of agitation of the duck, all other conditions being equal, are nearly the same. For a quantity of 0.1723 g of dimethylacetylenylcarbinol in a solution of 96% ethyl alcohol, the transition from the diffusion to the kinetic region occurs at 520 oscillations/min. With an increase in the quantity of the unsaturated compound (0.5168 g of dimethylacetylenylcarbinol) this transition takes place at 580 oscillations/min.

It would seem at first glance that increasing the quantity of the substance to be hydrogenated would facilitate an earlier transition from the diffusion to the kinetic region. Actually, however, experimental investigations have shown that when the quantity of dimethylacetylenylcarbinol is increased, the transition from the diffusion region to the kinetic takes place earlier. This becomes comprehensible if we consider not only the diffusion of the unsaturated compound but also the diffusion of hydrogen to the surface of the catalyst. The latter is apparently the limiting factor in the process of hydrogenation of a larger quantity of an unsaturated compound. Actually, molecules of hydrogen, before diffusing to the surface of the catalyst, must pass through two boundaries, gas-liquid and liquid-solid, while molecules of the unsaturated compound pass through only one boundary, liquid-solid. In

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the course of the reaction, therefore diffusion hindrances may delay the diffusion of hydrogen more effectively in comparison with the diffusion of the unsaturated compound. The rate of hydrogenation is evidently determined here by the rate at which hydrogen molecules approach the surface of the catalyst. This limiting factor, diffusion of hydrogen to the surface, can be overcome only by further increasing the intensity of agitation (in our case, up to 580 oscillations/min).

The hydrogenation of a 0.5163 g quantity of dimethylacetylenylcarbinol at a rate of agitation in the range between 520-580 oscillations/min can be thought to proceed in the kinetic region, with respect to the unsaturated compound, and in the diffusion range, with respect to hydrogen. At an agitation of 580 oscillations/min or higher, the limiting factor of hydrogen diffusion is overcome, and the reaction proceeds in the kinetic region, with respect to both components.

The next stage of the investigation was concerned with a study of the effect of the intensity of agitation of the reaction mixture on the rate of hydrogenation of dimethylacetylenylcarbinol (in water) in dependence on the quantity of catalyst. The experiments were conducted with 0.1723 g quantities of dimethylacetylenylcarbinol and 25 ml quantities of water distilled twice at 0°C. The results of this investigation are presented in Figure 5.

From an examination of the curves reproduced in Figure 5, it is evident that as the rate of agitation is increased from 210 to 580 oscillations/min, the rate of hydrogenation of dimethylacetylenylcarbinol in water increases, and the reaction proceeds in the diffusion region.

As the intensity of agitation is raised higher than 580 oscillations/min the rate constant of the hydrogenation ceases to depend on the rate of agitation, and the reaction proceeds in the kinetic region. In the diffusion region, particularly at low rates of agitation, an increase in the quantity of the catalyst has a small effect on the increase of the hydrogenation rate constant. In the diffusion region, there is a curvilinear dependence of the hydrogenation rate constant on the quantity of catalyst. In the kinetic region, the rate of hydrogenation of dimethylacetylenylcarbinol varies in direct proportion to the increase of the quantity of catalyst (Figures 5 and 6), and the ratio of the value of the rate constant in hydrogenation of dimethylacetylenylcarbinol $\frac{\Delta V_0}{\Delta t}$ cm³/min to the weight q of the Raney Ni catalyst, i.e., the specific catalytic activity for each experiment, remains constant, equal to approximately 16.4.

From the curves (Figure 5), it is easily noticeable that the transition from the diffusion region to the kinetic occurs at the same rate of agitation (580 oscillations/min), independent of the quantity of catalyst.

An increase in the reaction rate leads to an increased consumption of hydrogen on the surface, i.e., the diffusion of hydrogen to the surface of the catalyst becomes the limiting process in the course of the reaction. In this connection, it should be expected that in the hydrogenation of dimethylacetylenylcarbinol an increase in the quantity of the catalyst must cause the transition between the diffusion and kinetic regions to occur at higher agitation intensities. In our experiments this was not observed. To discover a satisfactory explanation for this phenomenon, it is necessary to turn to Figure 4. From Figure 4 it is evident that a decrease in the quantity of the substance to be hydrogenated displaces the boundary between the diffusion and kinetic regions in the direction of lower rates of agitation.

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In the latter case, it is necessary to take into account that the concentration of the substance being hydrogenated, relative to the quantity of catalyst, does not remain constant when the quantity of catalyst is increased. Therefore it is possible that within the known limits there begins to appear a compensation of some factors which retard the transition from the diffusion region to the kinetic and other factors which accelerate it.

Also of interest is the question concerning the influence of the nature of the solvent on the position of the diffusion and kinetic regions during the course of the dimethylacetylenylcarbinol hydrogenation reaction on Raney nickel.

We conducted no special investigation into the influence of the nature of the solvent. However, Figure 7 compares the rates of hydrogenation of dimethylacetylenylcarbinol in water and alcohol at 0°C and shows the positions of the transition of the diffusion region to the kinetic in each of these solvents. Figure 7 shows that the hydrogenation of dimethylacetylenylcarbinol proceeds more rapidly in alcohol than in water in both diffusion and kinetic regions. The transition from the diffusion region to the kinetic, when the hydrogenation is conducted in alcohol, occurs at lower rates of agitation (520 oscillations/min) than in water (580 oscillations/min).

The displacement in the direction of higher agitation intensities of the point of transition from the diffusion to the kinetic region, when dimethylacetylenylcarbinol is hydrogenated in water, is proof that under the given conditions the effect of the surface tension of water on the rate of diffusion of hydrogen to the surface of the catalyst begins to make itself felt. It is known that the value of surface tension for water is higher than the value for ethyl alcohol, while the solubility of such gases as H₂, N₂, O₂, etc., in various liquids decreases as the surface tension of the solvent is increased (9). It is therefore possible to assume on the basis of these properties of water and alcohol that a higher intensity of agitation is required to eliminate the effect of diffusion processes and pass over into the kinetic region when hydrogenation is conducted in water rather than alcohol.

Conclusions

1. A study was made of the kinetics of hydrogenation of dimethylacetylenylcarbinol in a solution of 96% ethyl alcohol and in water on a skeleton nickel catalyst.
2. It was established that, independent of temperature, quantity of the substance being hydrogenated, nature of the solvent (alcohol, water), and quantity of catalyst, the dimethylacetylenylcarbinol hydrogenation reaction proceeds according to the zero order.
3. Variation of the rate of agitation of the reaction mixture made it possible to delimit the diffusion and kinetic regions in the hydrogenation process.
4. In the diffusion region, the rate of the reaction depends on the intensity of agitation. In the kinetic region, the rate of hydrogenation is not dependent on the intensity of agitation.
5. It was established that when dimethylacetylenylcarbinol is hydrogenated in an alcohol solution at 0°C, the transition from the diffusion region to the kinetic occurs at lower rates of agitation (520 oscillations/min), while at 25°C, other conditions being equal, this transition occurs at higher rates of agitation (580 oscillations/min).

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6. In the diffusion region, the apparent energy of activation increases almost linearly as the intensity of agitation is increased.

In the kinetic region, the apparent energy of activation remains constant, equal to 4,733 cal/mole.

7. It was shown that when the quantity of the substance to be hydrogenated is increased, the transition from the diffusion to the kinetic region takes place at higher intensities of agitation. This phenomenon is evidently explained by a limiting factor in the process, the diffusion of hydrogen.

8. The rate of hydrogenation of dimethylacetylenylcarbinol in alcohol is almost three times as great as the rate of hydrogenation of the same substance in water, when other conditions are equal. The transition from the diffusion region to the kinetic, when dimethylacetylenylcarbinol is hydrogenated in alcohol, takes place at a lower intensity of agitation than in the case of hydrogenation in water.

9. In the diffusion region the rate of hydrogenation of dimethylacetylenylcarbinol on a skeleton nickel catalyst varies according to a concave curve as the quantity of the nickel catalyst is increased; in the kinetic region the reaction rate rises in proportion to the increase of the quantity of catalyst.

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[Appended figures follow.]

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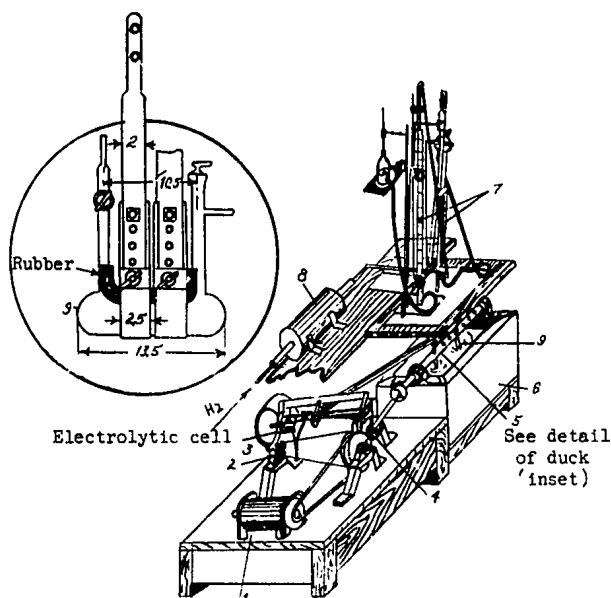


Figure 1. Apparatus for Catalytic Hydrogenation in the Liquid Phase. 1 - motor; 2 - first cone; 3 - second cone; 4 - eccentric; 5 - rod; 6 - iron tank for constant-temperature bath; 7 - measuring burette; 8 - electric furnace; 9 - duck.

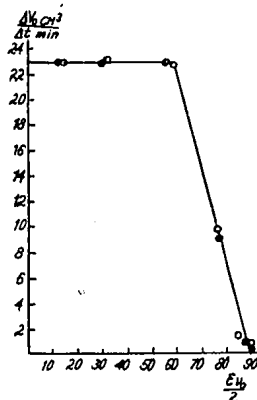


Figure 2. Variation of Rate of Hydrogenation Reaction With Time at 0°. The volume of reacting hydrogen is laid off on the axis of abscissae.

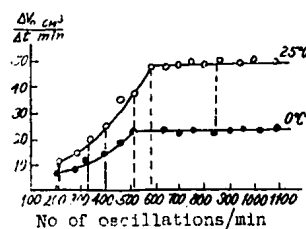


Figure 3. Effect of Rate of Agitation on Rate of Hydrogenation of Dimethylacetylenylcarbinol.

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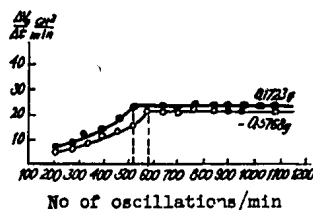


Figure 5. Effect of Rate of Agitation on Rate of Hydrogenation of Dimethylacetylenylcarbinol in Water. ● - experiments conducted with 0.3 g of Raney Ni; ○ - experiments conducted with 0.6 g of Raney Ni; ◐ - experiments conducted with 0.9 g of Raney Ni.

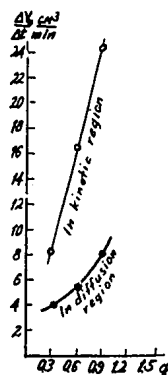


Figure 7. Dependence of Rate of Hydrogenation of Dimethylacetylenylcarbinol in Alcohol and in Water on Intensity of Agitation of Reaction Mixture

Figure 4. Dependence of Rate Constant of Hydrogenation on Intensity of Agitation of Reaction Mixture

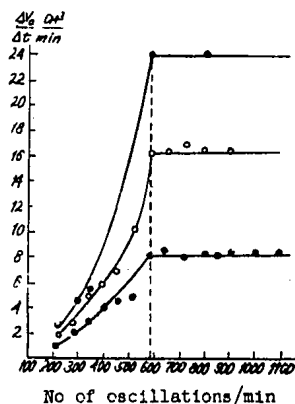
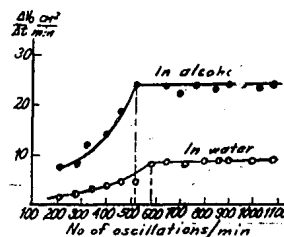


Figure 6. Dependence of Rate of Hydrogenation in Diffusion and Kinetic Regions on Quantity of Raney Ni Catalyst



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